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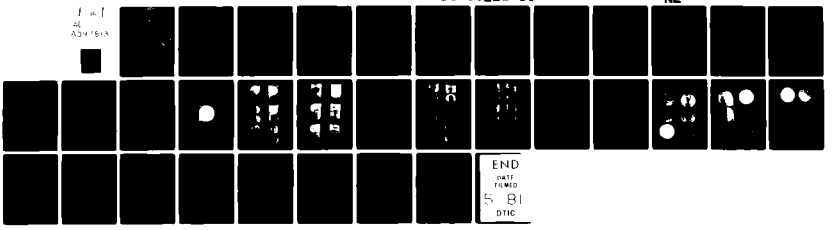

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NAVAL AIR SYSTEMS COMMAND MAINTENANCE TECHNOLOGY PROGRAM

SYSTEMATIC WEAR/CORROSION STUDY ANALYSIS OF ION PLATED SURFACES

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This study deals with the improvement in localized corrosion resistance and wear reduction of type M-50 steel used in engineering components such as bearings by use of overlay coating. Overlay coating materials selected for the improvement in localized corrosion (pitting) were titanium, chromium, nichrome (Ni-20Cr) and molybdenum deposited by physical vapor deposition techniques of direct evaporation (E), activated evaporation (AE), i.e. evaporation in the presence of a plasma, and ion plating (IP), and titanium carbide, titanium nitride and hafnium nitride coatings deposited by the activated reactive evapora-

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tion process (ARE).

Coated and control (uncoated) samples of M-50 steel were corrosion tested, the test designed to simulate the bearing environment inside an engine, the roller bearing-on-race geometry, as specified by Memorandum DTNSRDC TM 28-77-117. The test samples were first immersed for two hours in a polyester oil (test fluid) contaminated with 3 ppm chlorine in the form of sea water at 85°C, drip-dried for 30 minutes and then placed in the holding fixture so that a meniscus of contaminated oil was retained between the two parts. This system was then exposed to alternate cycles of moist air at 60°C ± 1°C in an oven for 8 hours and 3°C ± 2°C environment in a refrigator for 16 hours for a total period of two weeks. After the corrosion test, the samples were cleaned, the loose product wiped off, lightly polished and then examined under a microscope to observe the corrosion damage.

The results showed that the control (uncoated) samples were severly pitted along the contact line and general corrosion occured outside this region, whereas the samples with overlay coatings of titanium, chromium, nichrome (Ni-20Cr), TiC, and TiN completely eliminated the localized pitting corrosion. Coatings of these materials deposited by different techniques showed equally good results. Overlay coatings of molybdenum showed corrosion presumably due to the reaction with the sulfur content of the polyester oil. Hafnium nitride coating also showed pitting in the areas where the coating had peeled off the substrate after deposition and prior to the corrosion test.

Wear test samples with TiC and other hard coatings were prepared and submitted to NESO, San Diego for Rolling Contact Rod Wear Tests.

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I. OBJECTIVE

This contract calls for a review of critical aircraft wear components, selection of some of these components for potential wear reduction by ion plating/ion implantation and similar processes in cooperation with NESO personnel, application of the selected surface treatments and testing of the coated components.

II. IDENTIFICATION OF PROBLEM AREAS

As requested by the program sponsor, Mr. A. J. Koury of NAVAIR, Professor R.F. Bunshah visited NARF facilities at North Island, San Diego, on August 17, 1979, and met with David Stanley, Gary Kuhlman, Jeff Sakai and Phil Borja of the Materials Engineering Laboratory, Building 341, Code 340. A second visit was also made some weeks later. From these discussions, three projects were identified for investigation under this contract.

1. Localized Corrosion of type M-50 steel used in subcomponents such as bearings.

This is a serious problem costing in excess of $1 million for replacement of turbojet bearings alone. NRL is currently evaluating ion implantation to improve the pitting corrosion of AISI M-50 bearing alloy used in Navy turbojet engines. The worst corrosion problems occur in engines which experience intermediate usage and is exhibited by localized pitting at contact points between the bearing race and the rollers after engine shutdown. Ion implantation of corrosion resistant elements such as Cr at NRL shows that corrosion is significantly reduced in simulated field service corrosion tests with a salt-water contaminated polyester lubricant.(1)

Since ion implantation is a slow and costly process, it was decided that potentially faster and cheaper overlay coating techniques to improve corrosion

resistance would be investigated.

2. Wear Reduction of Engineering Components by Hard Overlay Coatings Using Rolling Contact Rod Wear Tests.

AISI M-50 steel rods were to be overlay coated with various hard compounds such as TiC, TiN, Ti_2N + TiN, etc., and tested by NADC in a Rolling Contact Fatigue Test machine.

3. Wear Reduction by Hard Overlay Coatings Under Oil Starvation Conditions

TiC or other hard coatings were to be deposited onto wear test blocks of CBS 600 material (Timken Company proprietory alloy) to be tested in a Timken Wear Test Device.

III. LOCALIZED CORROSION OF TYPE M-50 BEARING STEEL

A. Experimental Program

1. Selection of Coating Materials and Coating Techniques:

A number of coating materials which are known to have good corrosion resistance were selected for the present study to improve the localized corrosion resistance of M-50 bearing steel. The coating materials are titanium, molybdenum, chromium, nichrome (Ni-20Cr), titanium carbide, titanium nitride and hafnium nitride. These coatings were deposited in a vacuum chamber using an electron-beam heated evaporation source.

Several Physical Vapor Deposition techniques used to deposit single metal or alloy coatings were: (i) Direct Evaporation (E), (ii) Activated Evaporation (AE), i.e. evaporation in the presence of a plasma, and (iii) Ion Plating (IP). The compounds titanium carbide and titanium nitride were deposited by the Activated Reactive Evaporation Process (ARE). These processes are briefly defined below:

(i) Direct Evaporation: Direct evaporation is a process in which vapors

are produced from a liquid (or solid) source in vacuum where there is almost no surrounding gas. The escaping vapor atoms will travel in a straight line for some considerable distance before they collide with something; for example, the vacuum chamber walls, the substrate, or other vapor atoms. It is a line of sight process. The substrate is not biased and is usually at ground potential.

(ii) Activated Evaporation: This is a variation of direct evaporation carried out in the presence of a plasma; i.e. the vapor phase contains neutral atoms, energetic neutral atoms, positive ions and free electrons. The substrate is usually not biased and is at ground potential.

(iii) Ion Plating: Ion plating is a variation of the evaporation process in which the substrate is biased to a high negative potential which sets up a plasma due to an inert gas partial pressure (usually 10 to 50 μm) introduced into the chamber. Initially, the substrate and subsequently, the growing fiber is bombarded by the positive ions of the inert gas. A very small fraction of the evaporated metal flux is also ionized. The ion bombardment causes heating and microstructural rearrangements to occur in the deposited coating.

(iv) Activated Reactive Evaporation (ARE) Process: Activated Reactive Evaporation is a process for the deposition of refractory compounds by reaction between metal vapor atoms and molecules of a reactive gas (such as C_2H_2 or N_2 for the deposition of carbides or nitrides respectively). The reaction is aided by the presence of a plasma in the reaction zone (i.e. between the evaporation source and the substrate). The plasma is created by extracting the low energy secondary electrons from the plasma sheath above the molten pool by an electric field created by biasing an electrode placed above the molten pool to a small positive potential of 20 to 100 volts DC (or AC).

2. Materials Preparation and Coating Methodology

Rods of M-50 steel, 3/8" in diameter were supplied by Gary Kuhlman of NARF. Test samples of M-50 steel, 3/8" in diameter and 3/8" in height were cut from these rods. The faces of these cylindrical test samples to be coated and corrosion tested were polished using conventional surface preparation methods with the final alumina particle size being 0.05 μm. Duplicate samples were used for each coating material and each coating technique.

Figure 1 shows a schematic of the apparatus used for coating. To make a coating experiment, the samples were thoroughly cleaned, degreased and then mounted in a specially prepared substrate holder located just below the substrate heater. A base pressure of 10^{-5} torr or better was attained in the vacuum chamber and the samples were heated to approximately 350°C for 1/2 hour before evaporation and deposition of the coating material. Evaporation was accomplished by using an electron beam heated source, and the coating thickness was 5-7 μm. Samples were slowly cooled to room temperature before opening the vacuum chamber. Samples were examined for coating integrity and then used for corrosion testing.

3. Corrosion Simulation Test

Corrosion tests on the coated and control (uncoated) samples of M-50 steel were carried out following the procedure described in memorandum DTNSRDC TM 28-77-117. A copy is attached as Appendix I. This test is designed to simulate the bearing environment inside an engine. Figure 2 shows the arrangement of the test set-up. The cylindrical surface resting on the flat side of the upright cylinder is intended to simulate the roller bearing-on-race geometry. The cylinders were positioned in place and totally immersed for two hours in a polyester oil contaminated with 3 ppm chloride in the form of sea water. The two parts were then removed from the oil and allowed to drip dry for 30

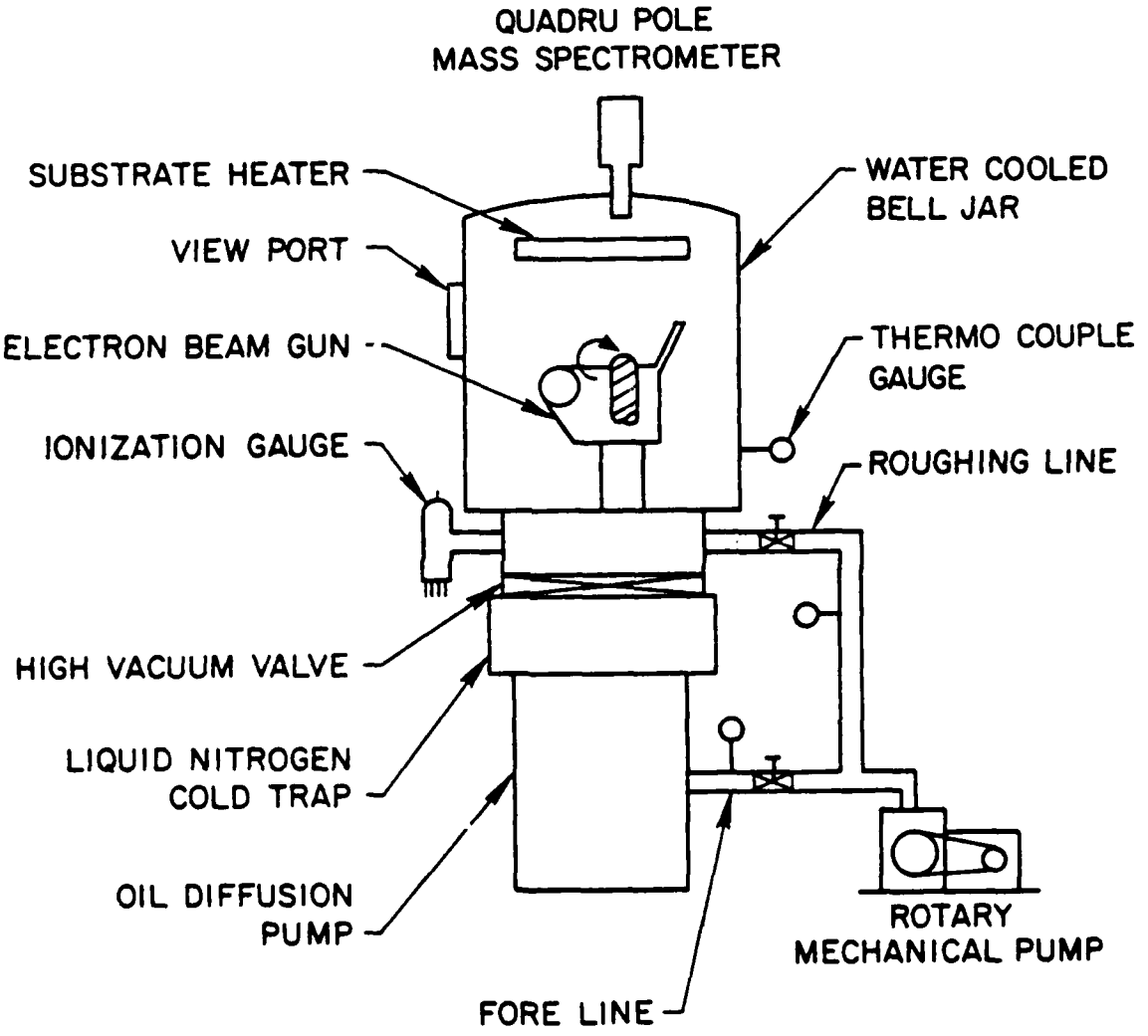
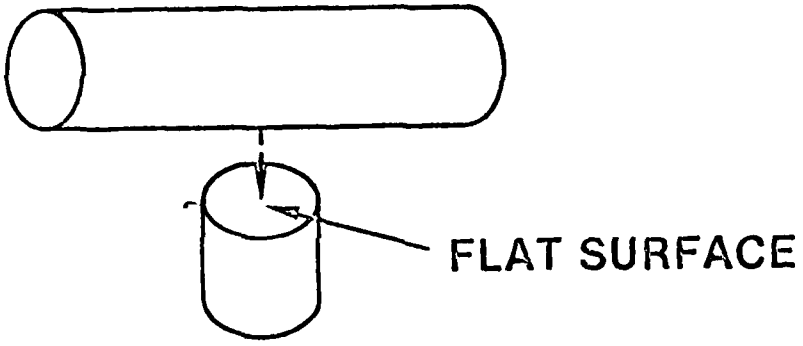


Figure 1: Schematic of the coating apparatus

1. Test pieces (both M50 alloy steel) were placed in contact as indicated by the dotted line.



2. Both pieces in place were immersed in chloride-contaminated oil for 2 hrs., removed, and allowed to dry.

3. A meniscus of contaminated oil was retained between the two parts:

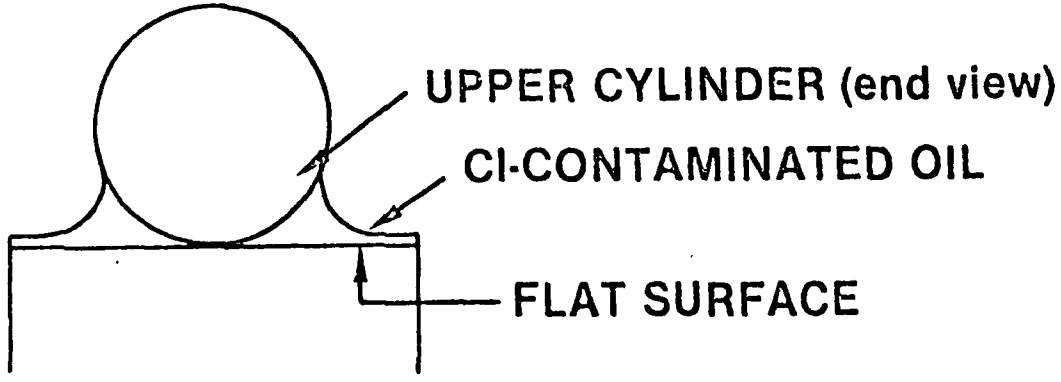


Figure 2 : Schematic of the corrosion test arrangement

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minutes before being placed in the holding fixture. A meniscus of contaminated oil was retained between the two parts, as shown in Figure 2. This system was then exposed to alternate cycles of moist air at 60°C ± 1°C in an oven for 8 hours and a 3°C ± 2°C environment in a refrigerator for 16 hours for a total period of two weeks. After the corrosion test, the samples were cleaned with benzene and acetone, and the loose corrosion product was wiped off the surface. The surface was then examined under a microscope.

B. Results and Discussion

Two separate corrosion test runs were carried out. The first corrosion test (11 samples) included the following samples: two control samples (uncoated), one sample each coated with titanium, molybdenum and nichrome with each coating material deposited by direct evaporation (E), activated evaporation (AE) and ion plating (IP). The results of this test were reported in Progress Report Number 1, dated April 17, 1980. It is believed that the polyester oil contaminated with chlorine (sea water) contained a somewhat higher level of chlorine than the recommended 3 ppm in the test procedure. This problem was corrected in the second corrosion test run (12 samples) which included the following samples: two control samples (uncoated), one sample each coated with TiC, TiN and HfN, two samples coated with chromium deposited by direct evaporation and activated evaporation, and duplicate samples coated with titanium, molybdenum and nichrome. Results of each corrosion test are presented separately in the form of photograph micrographs at 4x magnification of scattered light from the flat surfaces.

Figure 3 shows polished surface of a virgin (uncoated and uncorroded) sample. Figures 4 and 5 show the surfaces of control (uncoated) and coated samples after corrosion test runs 1 and 2, respectively. No polishing has been done on these samples after corrosion testing and therefore corrosion products are clearly

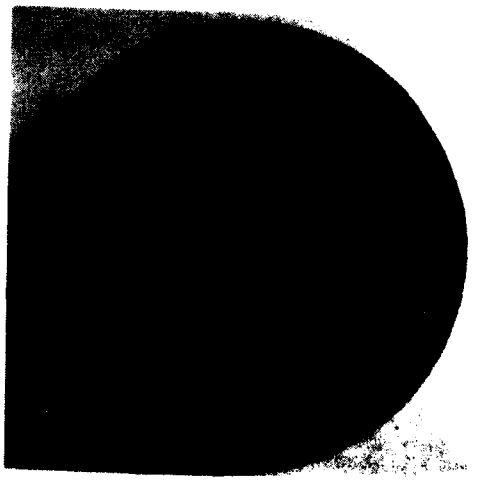


Figure 3 : Surface photograph of an uncoated and uncorroded M50 steel sample (Mag. X7)

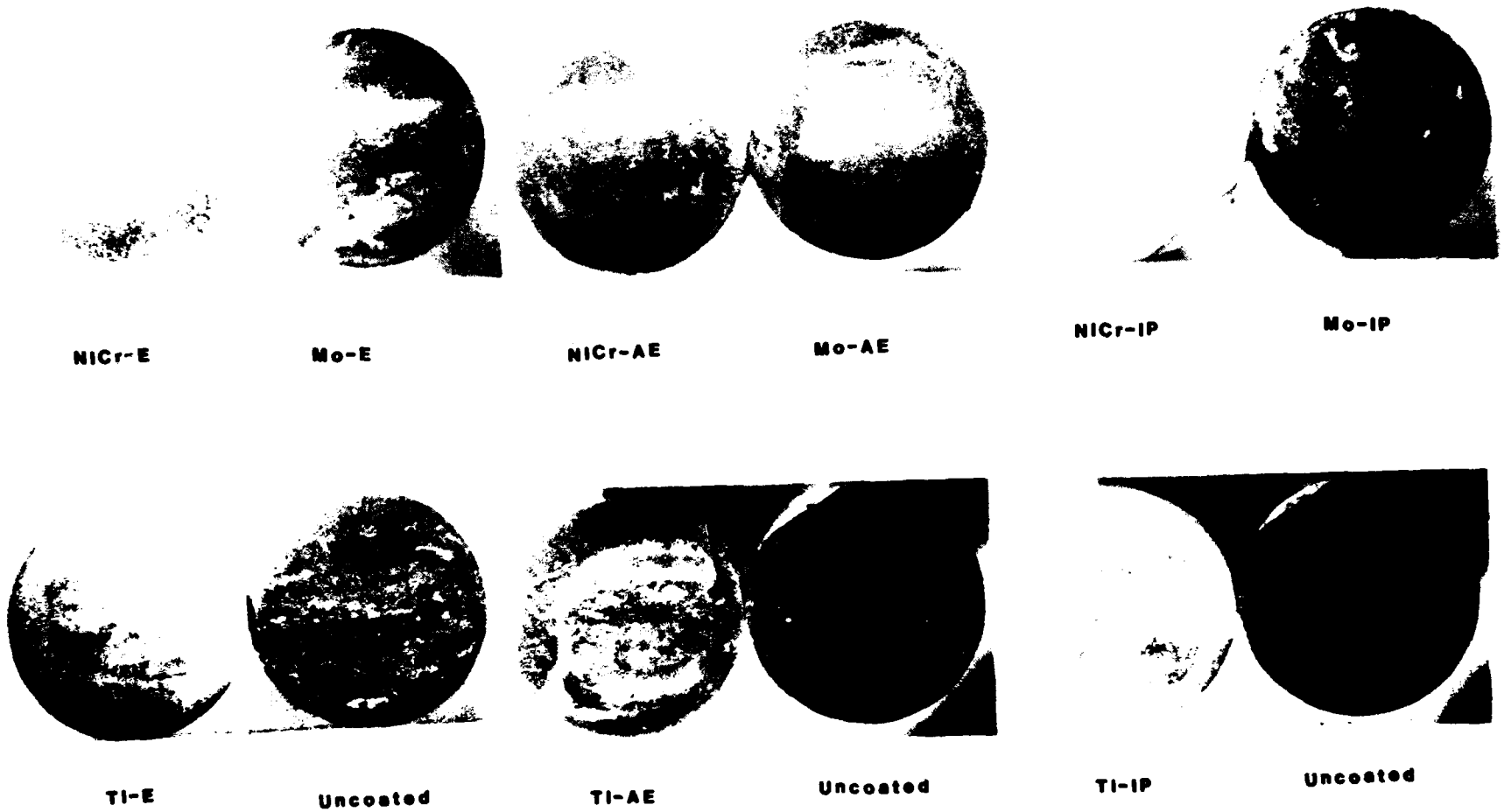


Figure 4 : Unpolished surface of uncoated (control) and coated samples after corrosion test run #1. (Mag. X4).

E: Evaporation, AE: Activated Evaporation, IP: Ion Plating, ARE: Activated Reactive Evaporation.

Figure 6 : Unpolished surface of uncoated (control) and coated samples after corrosion test run #2.(Mag. X4)

visible. All samples show a contact line on the surface which had developed from the cylindrical surface of the top sample during the corrosion test. Uncoated samples were completely covered with a "black" corrosion product and also showed a few deep pits on the surface. Most of the coated samples were partially covered with a corrosion product along both sides of the contact line, but no pits were observed on these surfaces. Titanium, chromium, nichrome and titanium carbide coatings by all the deposition techniques used in this investigation, were well-adherent and continuous on all samples, whereas ion plated molybdenum and hafnium nitride coatings had partially come off the surface and showed pits in the exposed areas. There was some damage observed at the edges of other molybdenum coatings and titanium nitride coating.

To observe the actual corroded surface, it was necessary to remove the corrosion product from all the samples. Therefore, the samples were lightly polished with 0.05 μm. Al_2O_3 grit in a slurry until only a few traces of corrosion product were left on the surfaces which ensures that the coating is not polished off the surface. Figures 6 and 7 show the polished surfaces of control (uncoated) and coated samples after corrosion test runs 1 and 2, respectively. The effect of corrosion can clearly be seen in these figures. For corrosion test run number 1, the control sample shows severely pitted and damaged area along the contact line. The attack generally occurs in two areas, e.g., a line of pits beneath the contact line between the cylindrical and flat surfaces, and general corrosion in the thin layer of oil outside this region. For corrosion test run 1, the uncoated sample shows severe corrosion whereas with the exception of one coated sample, none of the coated samples show any type of pitting or any other type of corrosion damage to the surface. The exception is the ion plated molybdenum sample where the coating had partially

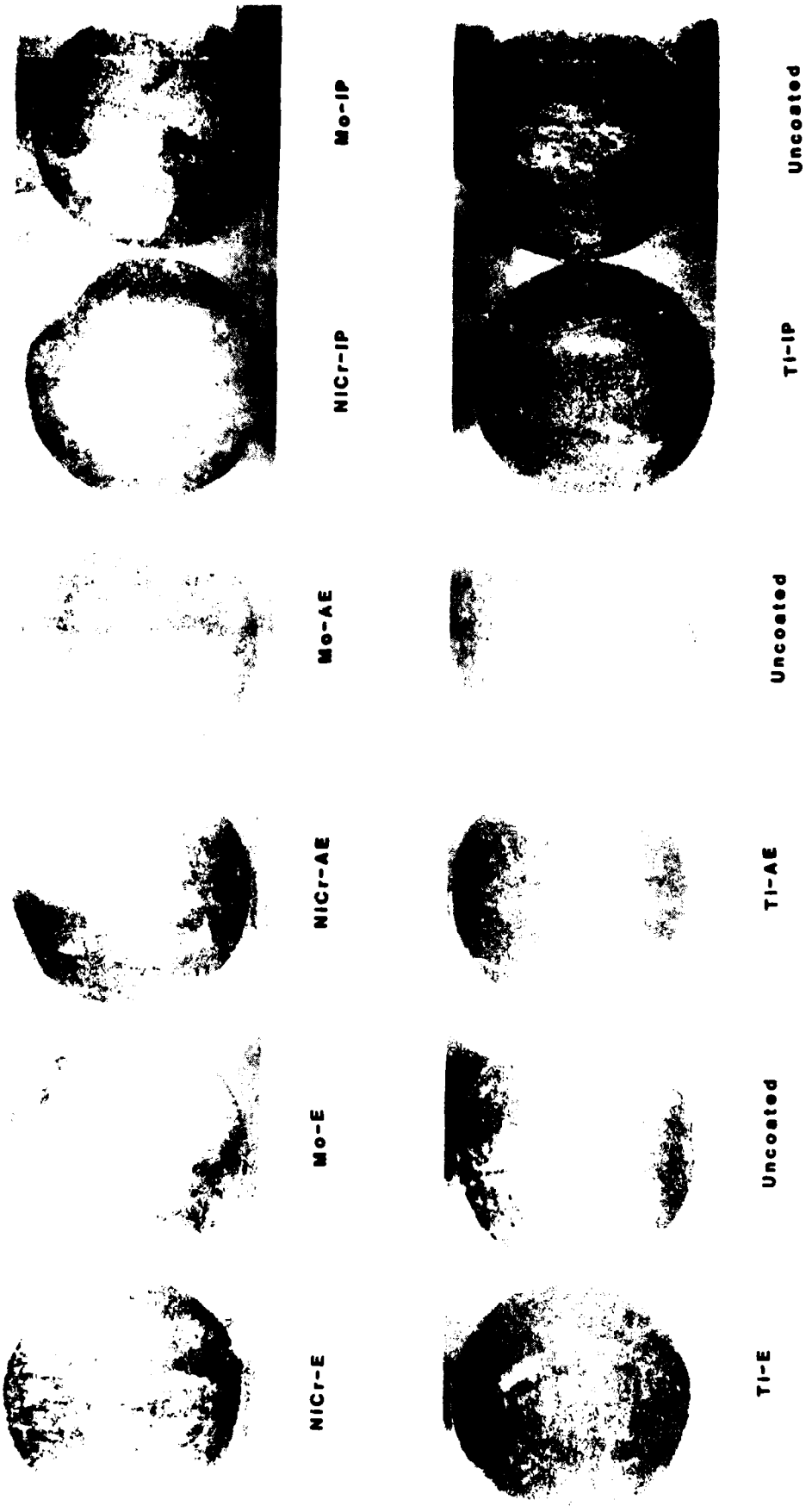


Figure 6 : Polished surface of uncoted (control) and coated samples after corrosion test run #1.(Mag. X4)

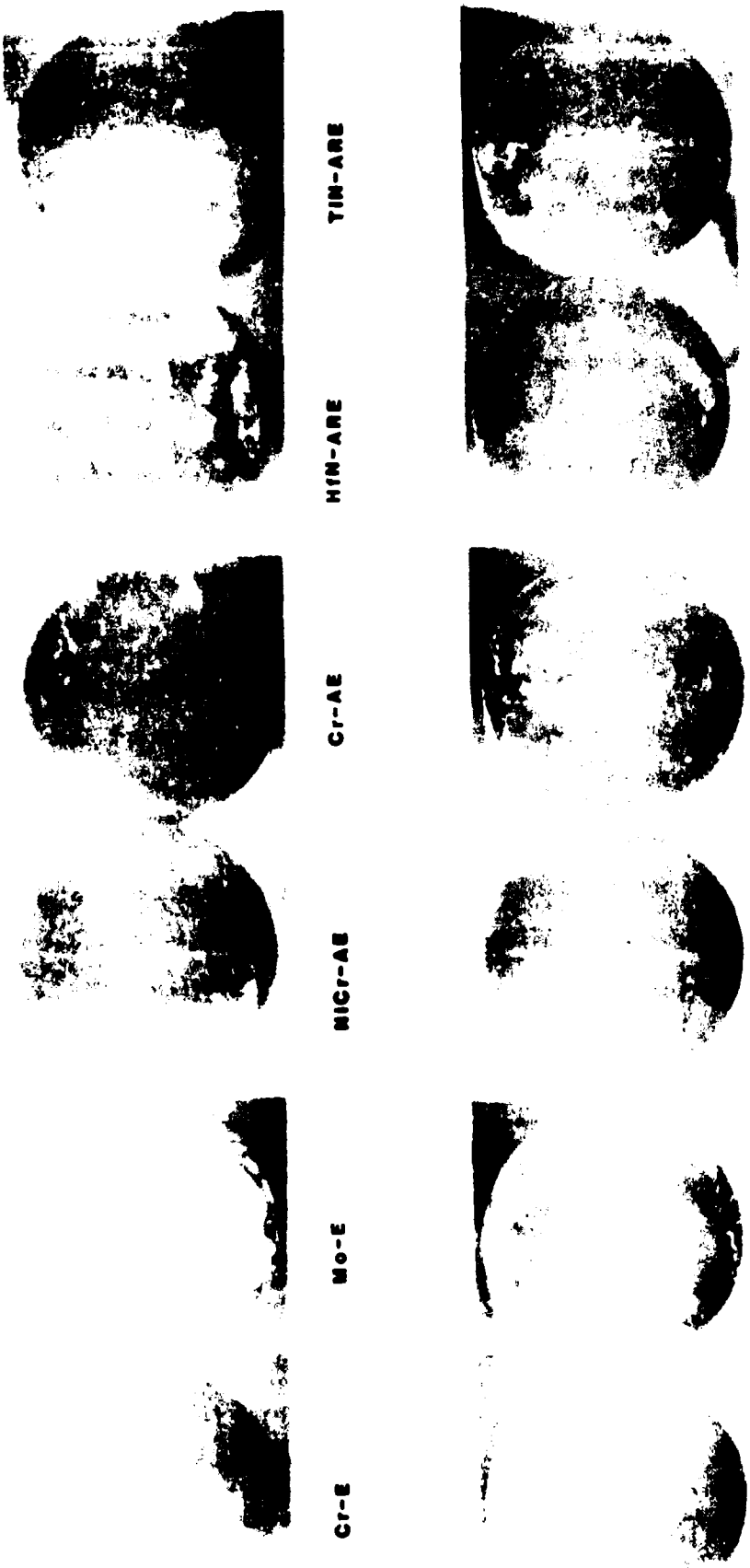


Figure 7 : Polished surface of uncoated (control) and coated samples after corrosion test run #2.(Mag. X4)

come off the surface. However, there is no pitting on the coating itself, but only in the exposed regions of the M-50 steel. This indicates that although molybdenum is resistant to corrosion, the coating either is not well-adherent to M-50 steel or is attacked by the lubricant fluid containing some sulfur compounds which presumably may react with the Mo coating. Titanium and nichrome coatings deposited by all three techniques do not show any type of pitting or any other type of damage. A contact line is observed on the soft nichrome coated sample (by direct evaporation) which is believed to be deformation markings from the cylindrical surface of the sample lying on top.

Figure 7 shows the polished surfaces of samples from corrosion test run 2. Again, the uncoated sample shows severe pitting along the contact line, and general corrosion on the rest of the surface. The damage is, however, not as severe as for the uncoated sample from corrosion test run 1. The reason for this is the higher amount of chloride (more than the recommended 3 ppm) in the test fluid for the corrosion test run 1. Samples coated with chromium, titanium, nichrome and titanium carbide show no corrosion damage. Titanium nitride coating also shows no corrosion, but has come off at the edges. The hafnium nitride coating has come off the surface at many areas and shows pitting in those regions. The hafnium nitride coating itself appears rough and very thick. It is not clear whether the hafnium nitride coating came off the surface due to its higher thickness or whether it is the corrosion attack. The coating performance results for corrosion test runs 1 and 2 are summarized in Table 1.

Figures 8 and 9 show the photographs of selected samples from the corrosion tests 1 and 2, at a magnification of seven times. These photographs clearly show the severe corrosion damage on uncoated samples whereas no pitting or any other type of corrosion damage on titanium, nichrome, chromium, titanium carbide and

Table 1: Summary of Localized Corrosion Test Results on M-50 Bearing Steel

Coating	Process	Corrosion Test Results
		Corrosion Test #1
Uncoated	-	Severe pitting corrosion along the contact line and general corrosion on rest of the surface.
Titanium	E	No corrosion.
Titanium	AE	No corrosion.
Titanium	IP	No corrosion.
Molybdenum	E	Coating damaged at edges but no pitting in coated area.
Mo	AE	Coating damaged at edges but no pitting in coated area.
Mo	IP	Coating peeled off. Sample corroded.
Nichrome	E	No corrosion but marking along the contact line due to deformation of the soft coating.
Nichrome	AE	No corrosion.
Nichrome	IP	No corrosion but marking along the contact line due to deformation of the coating.
		Corrosion Test #2
Uncoated	-	Pitting corrosion along the contact line and general corrosion on rest of the surface.
TiC	ARE	No corrosion.
TiN	ARE	No corrosion but slight damage at edges.
HfN	ARE	Corrosion attack in the areas where coating had come off, possibly due to the greater thickness.
Cr	E	No corrosion.
Cr	AE	No corrosion.
Ti	AE	No corrosion.
Ti	IP	No corrosion.
Ni-Cr	AE	No corrosion.
Mo-E		Coating damaged at edges.

E: Direct Evaporation
AE: Activated Evaporation
IP: Ion Plating
ARE: Activated Reactive Evaporation

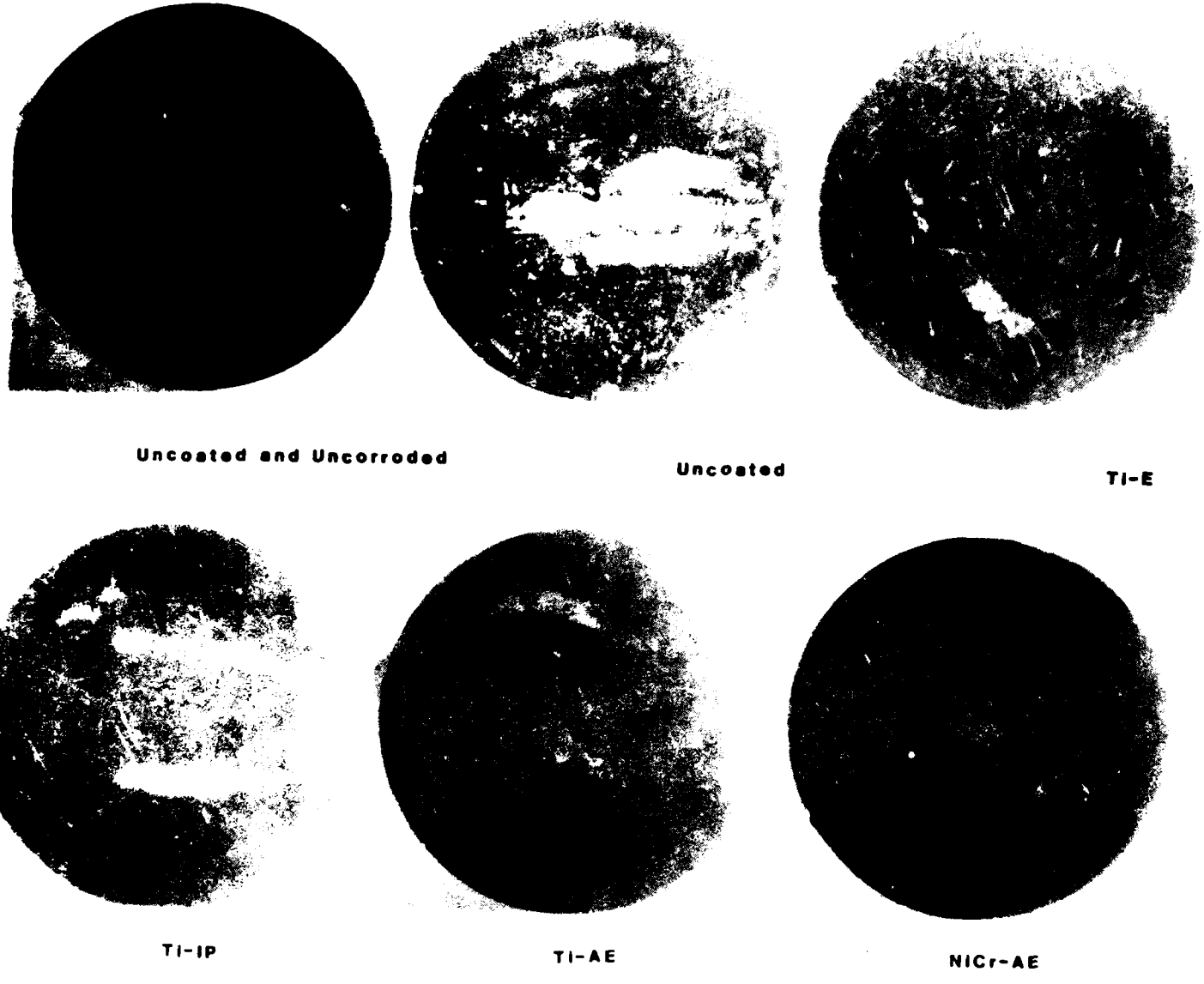


Figure 8 : Polished surface of uncoated and coated samples after corrosion test run #1.

All coated samples show no pitting. (Mag. X7)

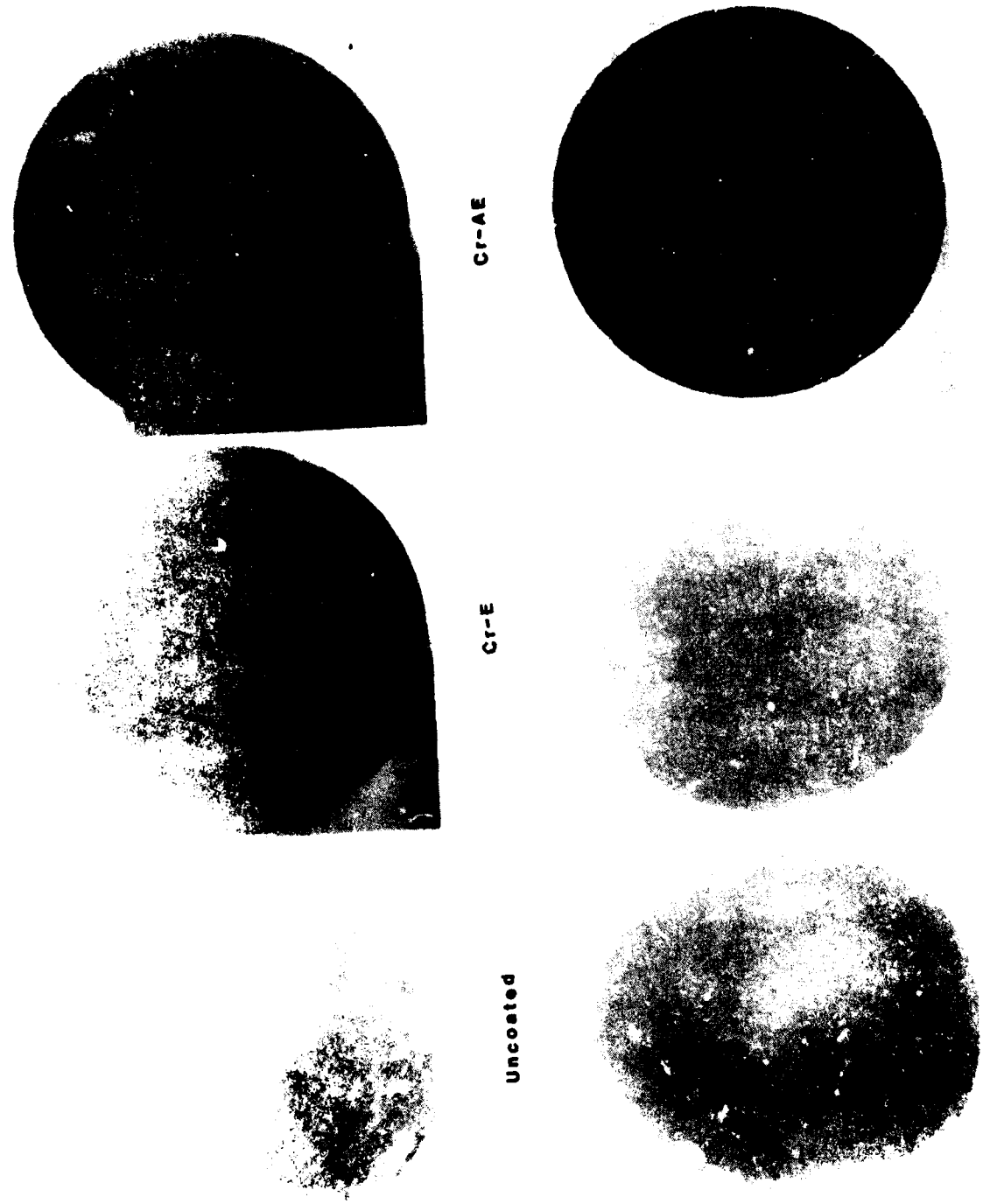
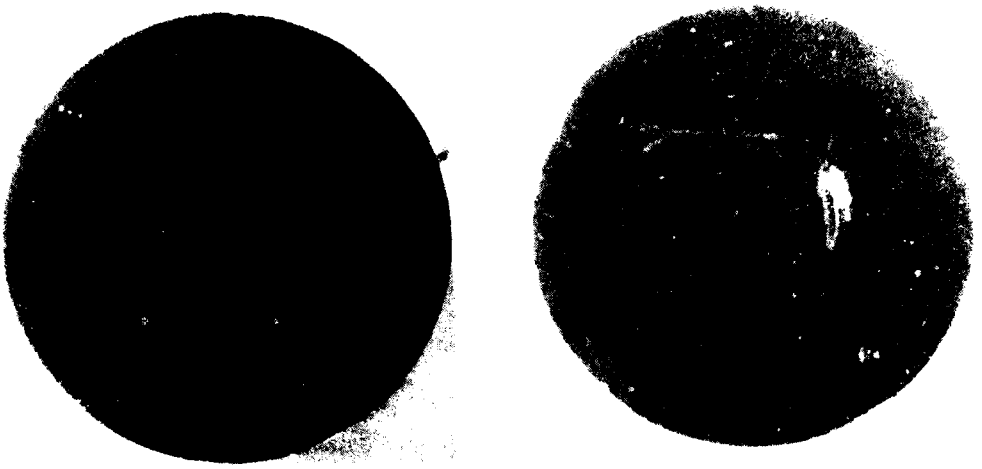


Figure 9 : Polished surface of uncoated and coated samples after corrosion test run #2.

All coated samples show no pitting.(Mag. X7)



TI-AE TI-IP

Figure 9 : continued

titanium nitride coated samples was observed.

C. Summary and Conclusions

Seven different overlay coatings were deposited onto flat surfaces of type M-50 bearing steel and subjected to a standard localized corrosion test as prescribed in Memorandum DTNSRDC TM 28-77-117 (Appendix I).

The results show that overlay coatings of titanium, nichrome (Ni-20Cr), chromium, TiC, and TiN completely eliminate the localized pitting corrosion of Type M-50 bearing steel in an environment of engine lubricant containing chloride ions. The coatings were deposited by three different physical vapor deposition techniques. Overlay coatings of molybdenum showed corrosion presumably due to the reaction with the sulphur content of the lubricant Hafnium nitride coating also showed pitting in the areas where the coating had peeled from the substrate after deposition and prior to the corrosion test.

These data show that coatings of Ni-20Cr, Ti, Cr, TiC and TiN inhibit localized corrosion in the selected environment and test conditions equal to the results obtained by ion implantation of Cr.[2]

Since the TiN and TiC hard overlay coatings reduce wear as well as improve corrosion resistance, this represents a promising new development over ion implanted materials from two viewpoints. One, overlay coatings are effective in reducing wear even in dry (i.e. unlubricated) or starvation lubrication conditions, whereas ion implantation is not effective under these conditions.[3] Second, overlay coating processes are currently much less expensive than ion implantation.

A further point to be considered is as follows. As stated by Hirvonen and Butler[1], it is generally believed that coatings reduce the fatigue life of components whereas ion implantation does not. This now has been shown to be incorrect since ion-implanted noble metal coatings and overlay hard coatings

(ref. T. Spalvins NASA-Lewis)[4] increase fatigue life. The important factor to be considered is whether the surface treatment (i.e. overlay coating, diffusion coating or ion implantation) produces tensile or compressive residual stresses on the surface. If they are tensile stresses, the fatigue life will decrease and if they are compressive the fatigue life will increase. A proper choice of coating parameters can give compressive stresses. Hence this problem should be recognized and used to advantage. The case can therefore be made for further studies on corrosion resistance and fatigue behavior of components using cheaper overlay coatings as compared to the more expensive ion implantation process.

IV. WEAR REDUCTION OF ENGINEERING COMPONENTS BY HARD OVERLAY COATINGS USING THE ROLLING CONTACT ROD WEAR TESTS

Four rods of Type M-50 high speed steel 6 inches long were coated on the cylindrical surface for a length of 3 inches. The coatings were deposited using the ARE process. The coating composition and thicknesses are:

	Composition	Thickness
1.	TiC	5-6 µm.
2.	TiC/Ti/TiC (multiple layers)	5-6 µm.
3.	TiC	1-2 µm.
4.	TiN	5-6 µm.

The coated rods have been forwarded to Mr. Gary Kuhlman, NARF, NAS, North Island, San Diego, for testing.

V. WEAR REDUCTION BY HARD OVERLAY COATINGS UNDER OIL STARVATION CONDITIONS

TiC coatings 4-5 µm. thickness were deposited onto two test blocks of case carburized CBS 600 steel on two adjacent surfaces at a deposition temperature of 180°C using the ARE process. They were tested by the Timken Company with the Timken Lubricant and Wear Test Machine using an oil-off scoring resistance

procedure. The results showed no improvement for the coated versus the untreated surface[5]. The report from Timken indicated that the coating deposited at 180°C was non-uniform in comparison to a previous coating where the deposition temperature was 540°C. It is difficult to draw many conclusions from a single test run. Obviously, if a lower deposition temperature is necessitated by the substrate material tempering parameters, the coating parameters and thicknesses must be varied. While this single test yielded poor results, other data on improvements in friction and wear using hard overlay coatings[3,4,6] justifies a more detailed study in the future.

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3. A.K. Suri, R. Nimmagadda and R.F. Bunshah, "Application of Ion Implantation and Ion Plating on Stainless Steel, Titanium and Aluminum," U.C.L.A., December 1978. Report No. NADC-79128-60. Also published in Thin Solid Films, Bul. 64, Page 191 (1979).

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5. P.S. Orvos, "Wear Tests of Overlay Coated (TiC) CBS 600 Steel," The Timken Company Physical Laboratory Report, Test No. 281.3.0, Supplement 1, May 1980.

6. T. Jamal, R. Nimmagadda, and R.F. Bunshah, "Friction and Adhesive Wear of Titanium Carbide and Titanium Nitride Overlay Coatings," presented at the International Conference on Metallurgical Coatings, April 21-25, San Diego, California. To be published in Thin Solid Films, 1980.

A P P E N D I X A

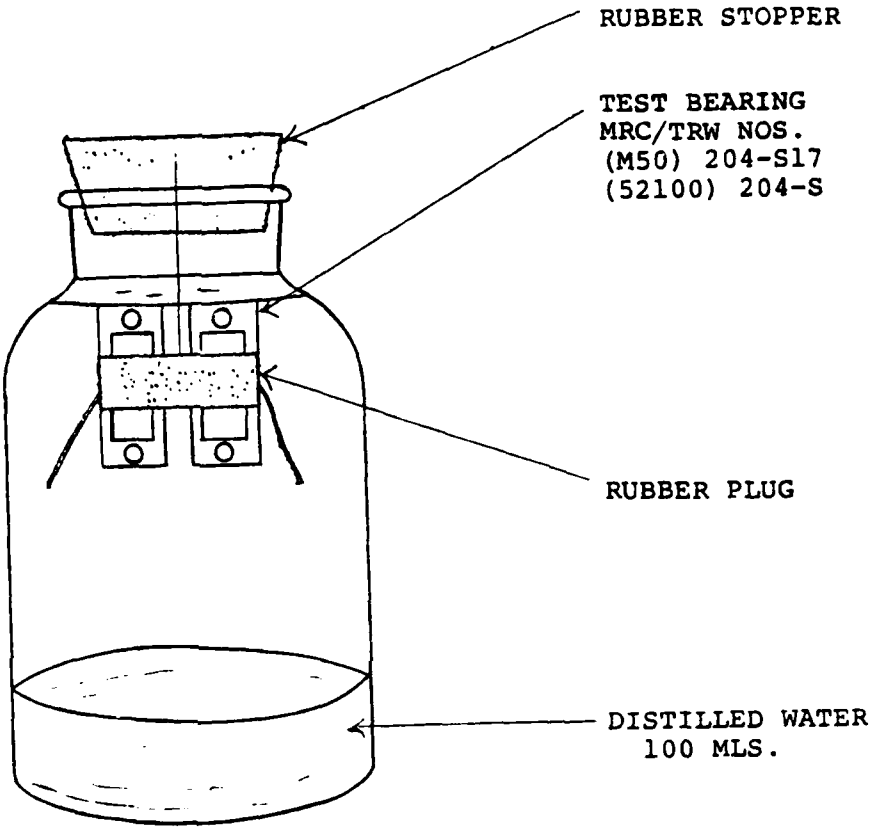
CORROSION EXPERIMENT METHOD

DTNSRDC TM 28-77-117

Corrosion Experiment Method

Each experiment is started on a Monday morning. The corrosion cell is a one litre glass bottle with a rubber stopper covered with polyvinyl plastic film. A 100 ml portion of distilled water is added to each cell. The bearing to be used is cleaned with successive washings in two baths each of benzene, naphtha, and acetone, in that order. It is air dried, then immersed in the fluid under evaluation at 83°C, with hand rotation every ten minutes for a two-hour soak period. Prior to use, 3 p/m of chlorides are added to the fluid under evaluation as ASTM D-665 seawater; then the water content of the fluid is adjusted to 600 p/m total water by addition of distilled water. The bearing is then drained 30 minutes at ambient temperature and then suspended, using a corrosion resistant wire, from the rubber stopper over the water in the cell. The bottle is closed and the cell placed in an oven for the start of a 14-day exposure period. During this period the cell is temperature cycled every 24 hours in a 60°C $\pm$ 1°C oven for 8 hours and in a 3° $\pm$ 2°C refrigerator for 16 hours. The cells remain in the oven during each of two weekends, with the oven heater shut off at 1600 on Friday and turned on the 0800 on Monday. After the initial 24 hours of the exposure period, the distilled water is replaced with a fresh amount of distilled water. After the 14-day exposure period, the bearings are disassembled. The races and balls are prepared for inspection by cleaning with benzene and acetone and loose products of corrosion are wiped off of bearing surfaces. The number of pits of 0.01" diameter, or larger, are counted at 15 x magnification.

CORROSION TEST CELL



1 LITER CELL

NAPTC-WUA-782

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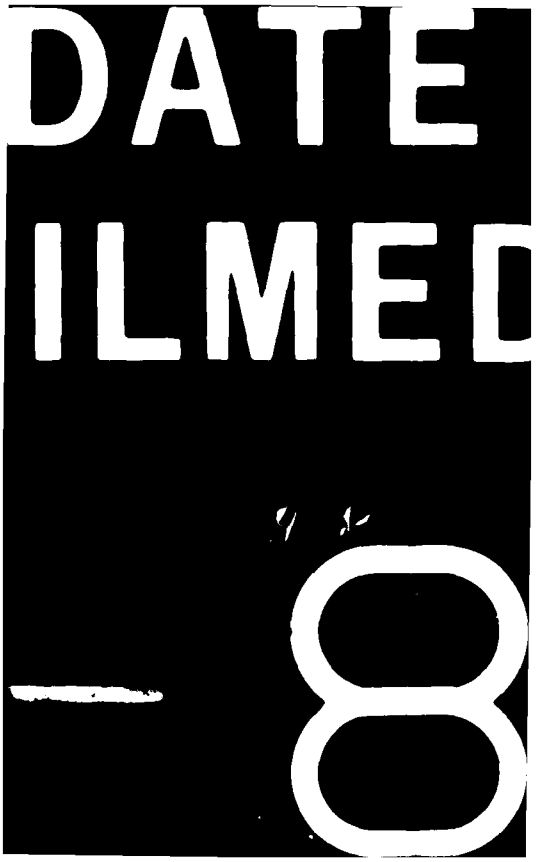
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